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LEAR SIEGLER, INC.

RESEARCH LABORATORIES

SANTA MONICA, CALIFORNIA

511 750



SEMICONDUCTOR THIN FILMS

CONTRACT NO. AF33(657)-7623

**Aeronautical Systems Division
Wright-Patterson Air Force Base, Ohio**

QUARTERLY REPORT NO. 4
August 1, to October 31, 1962

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1.0 PLANS FOR THE FOURTH QUARTER

Work on the project was to have been finished this quarter and a final report written. However, uncertainty about an extension of time and, therefore, the form of a next report has delayed the fourth quarter report until now. The final report will be submitted in April.

1.1 Original Plans for the Fourth Quarter

- 1.1.1 To construct a new source boat designed to prevent projection of GaAs particles.
- 1.1.2 To deposit GaAs on BeO.
- 1.1.3 If CeO₂ is delivered, to deposit GaAs on CeO₂.
- 1.1.4 To investigate the effect of rate of deposition by depositing from 3 cm and 10 cm distances.
- 1.1.5 To investigate the effects of an extended (1/2 hour) preheating of the substrate and of a helium flush of the deposition chamber before deposition.
- 1.1.6 To investigate the effect of annealing on crystallinity, resistivity, and type of the deposits by slow cooling from about 800°C. If the type changes, to attempt to make a diode based on the process.
- 1.1.7 If time is available, to construct a completely evaporated point contact diode based on a controlled area and position of the contact.



1.2 Additional Plans for the Fourth Quarter

- 1.2.1 To set up for and measure Hall mobilities at room temperature.
- 1.2.2 To investigate the possibility that the acid treatment used in peeling a film selectively dissolves part of the film.
- 1.2.3 To investigate the possibility that electron beam heating in the microscope significantly alters the films.
- 1.2.4 To set up for insulator deposition onto GaAs films.
- 1.2.5 To borrow or start building an ultra high vacuum system in which to deposit films.
- 1.2.6 To re-examine old films with the improved resolution available in the electron microscope.
- 1.2.7 To investigate barrier and/or capacitive contacts to the films as possible methods of device construction.

2.0 ACCOMPLISHMENTS

Dr. Milo Macha, a new member of the Research Laboratories is available part time for consultation and experimental work on the semiconductor thin film projects. Dr. Macha received his Bachelor and Ph.D. degrees from the Charles University of Prague, Czechoslovakia. He has worked in the fields of research and development in ferro-electric materials, pilot production of silicon controlled rectifiers, silicon solar cells, and controlled



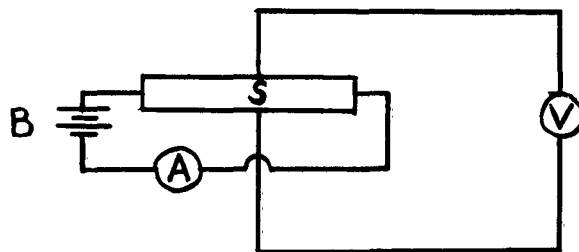
rectifiers, process control in pectin production, and as a consultant to Georgian China, Ltd., Canada, and to N.V. Naga Hidjau, Indonesia. He has published papers on porcelain insulators, light-weight ceramics, electrical kilns for ceramic industry, and irregular drafts in annular kilns. Dr. Macha has U.S. patents on Protective Coating for Silicon Diodes and Ruggedized Contact on Silicon Solar Cells. His affiliations are IRE, Acoustical Society of America and American Ceramic Society.

- 2.1 A new flash evaporation source was made from 3/8 inch tantalum tubing by pinching the ends and cutting holes in the periphery (Figure 1). Powder falls through a funnel into one hole and vapor emerges from the other hole to deposit on the substrate. In the first try, many fewer GaAs particles stuck to the substrate than has been the case using an open boat and these may have come from the entry port which was not shielded from the substrate. A photograph by transmitted light of a portion of the 2500A deposit (Figure 2) shows no thin spots and may be compared with Figure 6.
- 2.2 Bridge shaped deposits of p GaAs (J529) and n GaAs (J539) were deposited for trial Hall measurement. The deposits are relatively free of thin spots but are thinner for the same amount of source material than if deposited from an open boat.

2.3 Deposition of more GaAs films was deferred in favor of investigating the deposition of Si on Si, on BeO, and on SiC. No deposition rate, annealing, preheating, or helium flush experiments were made.

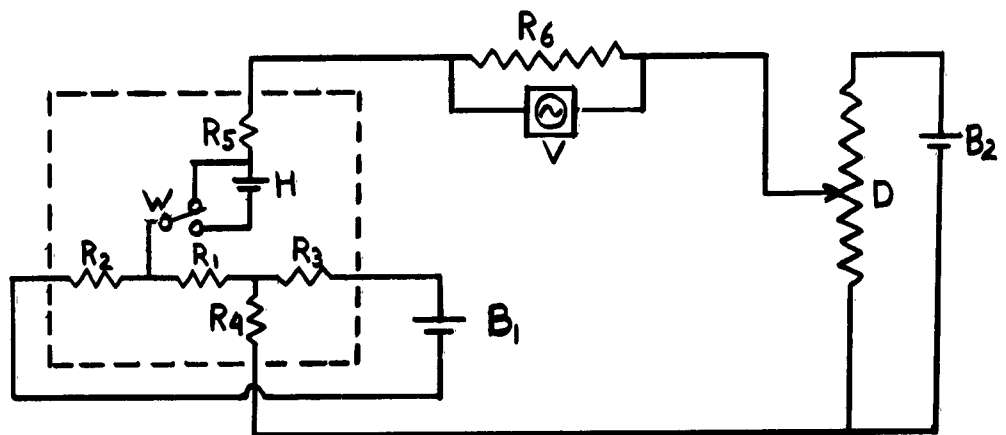
2.4 The physicist due to report in August accepted an offer elsewhere. However, Lawrence N. Hamet was hired to set up equipment for and to make measurements on our thin films. Mr. Hamet received his B.S. Metallurgical Engineering Degree from the University of Arizona. He has also taken graduate courses at the University of Arizona, Carnegie Tech, and UCLA. His experience includes work in the field of development of n and p type gold alloys, development of germanium dendrites, design and development of high conductance microdiodes by n⁺ diffusion and gettering and of low capacitance diodes by using preferential etching at the junction, and the development of integrated circuits.

Since October 1st, he has set up apparatus for Hall measurement according to the following basic system:



The current through the sample, S , in the presence of a magnetic field normal to the plane of the sketch, generates a voltage normal to both the current and the magnetic field which is measured by V .

Slight electrical misalignment of the contacts which receive the voltage for V is practically unavoidable because the films do not have a uniform thickness. The equivalent circuit actually used is therefore as follows:



Where $R_2 + R_1 + R_3$ represents the resistance of the sample to the generating current driven by B_1 , R_1 being the resistance along the sample between misaligned contacts; $R_4 + R_1 + R_5$ is the resistance across the sample in series with the generated Hall voltage represented by battery H . W is a switch which, with H , represents the effect of turning on the magnetic field. B_2 is a battery which, with Decavider,

D, balances the misalignment and/or Hall voltages. The null readings are taken across the 1 megohm resistor R_6 by oscilloscope V. D is adjustable to 10,000 ohms in 0.01 ohm steps; V has a maximum sensitivity of 200 microvolts per centimeter. The magnet is being calibrated now by measurement of the Hall voltage generated by a calibrated Beckman Model 350 indium antimonide thin film Hall generator.

- 2.5 Part of a 3000A GaAs deposit was peeled, part was replicated, and part was immersed in 1-1 HCl and then replicated. The 3000A thick film shows a finer structure than do thinner films (Figure 3, and Figure 6 of Quarterly Report No. 3). Figures 4 and 5 indicate that there was some etching of the film by immersion in HCl. Figure 6 shows that immersion in HCl peeled some spots on the film and part of the edges, but there are still thin areas in the acid treated film and its thickness has not decreased.
- 2.6 GaAs films were left in the strongest electron beam of the electron microscope for up to 30 minutes and changes in the film photographed. Figure 7 shows that the film's opacity increases with exposure and there is a smaller crystallite size in the heated film, but the pattern of thin spots and holes does not change; no area in Figure 7a is less transparent than the same area

in Figure 7b. Figure 8 shows the increase in opacity and, again, no change in the arrangement of pinholes and thin areas. Films left in the beam for a still longer time evaporate almost completely leaving a very thin film (probably a carbonaceous deposit) with scattered opaque specks.

- 2.7 No deposits of insulator were made.
- 2.8 A small ultra high vacuum system will be constructed in our laboratory, as no system could be found which was available for our use. First thoughts are to add to our present system a small chamber with fewer or no organic seals and an additional pumping stage, such as an absorption or ionic pump.
- 2.9 Electron micrographs at 35,000X had shown the dislocations and/or stacking faults in GaAs films but were not detailed enough to show them in Ge deposits. The microscope was therefore set up for higher magnification and better resolution by insertion of smaller opertures and the stigmator.

Micrographs made of an old Ge film using the new arrangement showed at 35,000X, a similar arrangement of dislocations and/or stacking faults (Figure 9) to that in older micrographs of GaAs films, such as Figure 7a. Micrographs of a GaAs film now show the arrangement of defects more



clearly (Figure 10).

Though 20A is the best resolution guaranteed by Phillips for the EM100B, copper phthalocyanine, which has an interplaner spacing of 12.5A, will be recrystallized and examined in the hope of getting a resolution of 12.5A.

- 2.10 Work with silicon was done on another contract. Satisfactory conditions were found for etching SiC and BeO and deposits up to 2 mils thick were made. The surfaces of the deposits are similar to those of Ge and GaAs deposits. One polycrystalline deposit peeled spontaneously and examination in the electron microscope showed a coarse structure similar to that of Ge. X-ray patterns of single crystal deposits had the same diffuse X shown by patterns of GaAs and Ge deposits.

An effort to make a fused alloy junction in a thin film of Si was unsuccessful. Thin layers of gold-antimony and aluminum were deposited as small areas on a film of Si on Si at substrate temperatures below the eutectic temperature and subsequently fused in vacuum. The I-V characteristic was that of a resistor (Figure 11).

The alternate approach of forming rectifying contacts by surface oxidation of the semiconductor film and then depositing contacts was successful both with Si and GaAs films. Si, n type, 200 ohm centimeter, was oxidized in



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- 3.3 To make Hall mobility measurements at room temperature and to set up for low temperature measurement.
- 3.4 To deposit GaAs on BeO and continue the search for CeO₂ and other substrates.
- 3.5 To continue improvement of electron microscopy techniques.
- 3.6 To continue improvement of the GaAs evaporation source.
- 3.7 To investigate deposition at microns per second.
- 3.8 To continue device exploration using films of available quality.

4.0 LIST OF ILLUSTRATIONS

Figure 1 - Tantalum tube flash evaporation source.

Figure 2 - Micrograph by transmitted light of 2500A GaAs film J529C.

Figure 3 - Transmission electron micrograph of 3000A GaAs film J495C4, 10,000X.

Figure 4 - Electron micrograph of replica (polystyrene) of 3000A GaAs film J495D as deposited, 10,000X.

Figure 5 - Electron micrograph of replica (polystyrene) of 3000A GaAs film J495D after immersion in 1-1 HCl for 30 minutes, 10,000X.



Figure 6 - Micrograph by transmitted light of 3000A GaAs film J495D which was broken and half (on left) immersed in 1-1 HCl for 30 minutes, 50X.

Figure 7a Transmission electron micrograph of 700A GaAs film J489-1, 35,000X.

Figure 7b Same area after exposure for 30 minutes to the strongest electron beam available in the microscope.

Figure 8 - Transmission electron micrographs of 3000A GaAs film J464 after (a) 5 minutes, (b) 6 minutes, and (c) 15 minutes in the strong electron beam.

Figure 9 - Transmission electron micrograph of 1500A Ge film J392 peeled from CaF_2 , 35,000X.

Figure 10a Transmission electron micrograph of 1500A GaAs film J410 peeled from CaF_2 , 35,000X.

Figure 10b 90,000X.

Figure 11 I-V characteristic of Si film on bulk Si with evaporated, then alloyed, contacts of Al and Au-Sb. 0.05 ma/div. vertical, 0.5 v/div. horizontal. J534.

Figure 12 I-V characteristic of 5000A p type Si film on BeO , oxidized 1 hour at 900°C . 6 square mil Au vs. 2000 square mil Al contacts. 0.05 ma/div. vertical, 2 v/div. horizontal. J539.

Figure 13 I-V characteristic of 5000A n type GaAs film on CaF_2 , oxidized 1 hour at 550°C . 6 square mil Sn vs. 10,000 square mil Sn contacts. 0.05 ma/div. vertical, 0.2 v/div. horizontal. J530B.

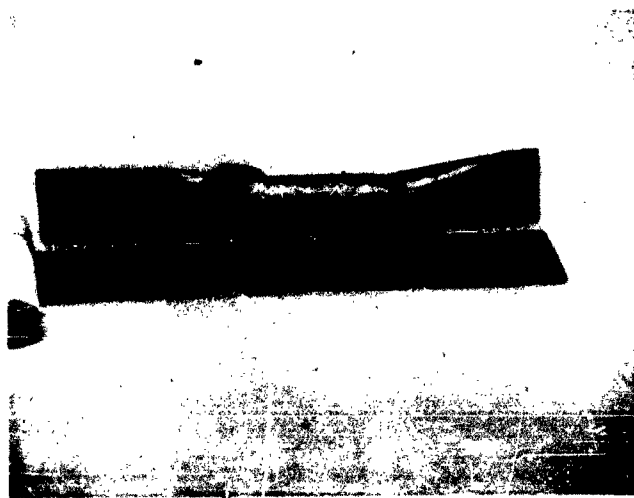


Figure 1
Tantalum tube flash evaporation source.



Figure 2
Micrograph by transmitted light of 2500A GaAs film J529C.

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Figure 3

**Transmission electron micrograph of 3000A GaAs film
J493C4, 10,000X.**

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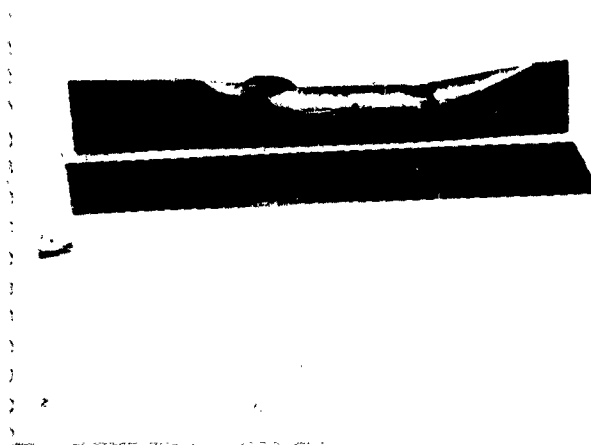


Figure 1
Tantalum tube flash evaporation source.

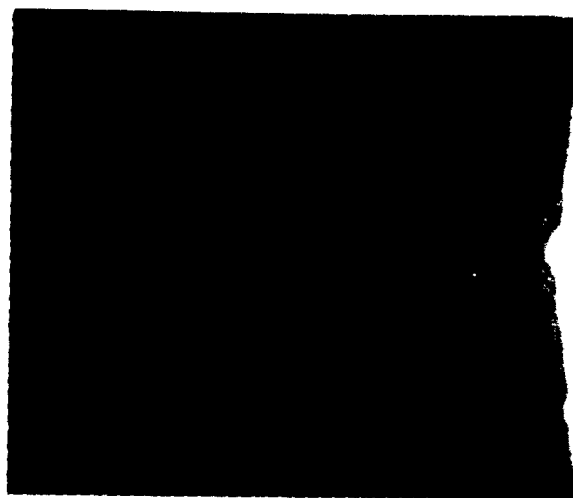


Figure 2
Micrograph by transmitted light of 2500A GaAs film J529C.



Figure 3

**Transmission electron micrograph of 3000A GaAs film
J495C4, 10,000X.**

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Figure 4

Electron micrograph of replica (polystyrene) of 3000A GaAs film J495D as deposited, 10,000X.



Figure 5

Electron micrograph of replica (polystyrene) of 3000A GaAs film J495D after immersion in 1-1 HCl for 30 minutes, 10,000X.

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Figure 6

Micrograph by transmitted light of 3000A GaAs film J495D which was broken and half (on left) immersed in 1-1 HCl for 30 minutes, 50X.

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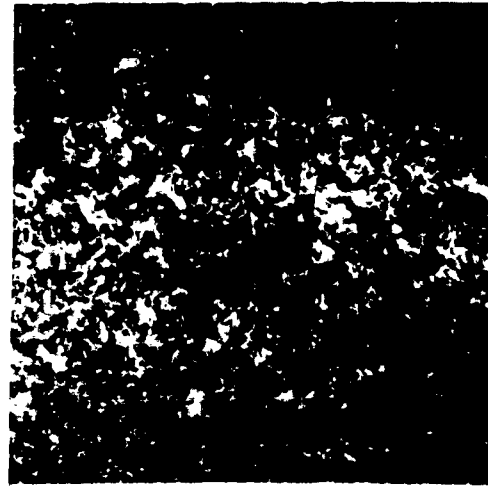


Figure 7 (a)

Transmission electron micrograph of 700A GaAs film
J489-1, 35,000X.



Figure 7 (b)

Same area after exposure for 30 minutes to the strongest
electron beam available in the microscope.

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(a) (b) (c)

Figure 8

Transmission electron micrographs of 3000A GaAs film J464 after, (a) 5 minutes, (b) 6 minutes, and (c) 15 minutes in the strong electron beam.

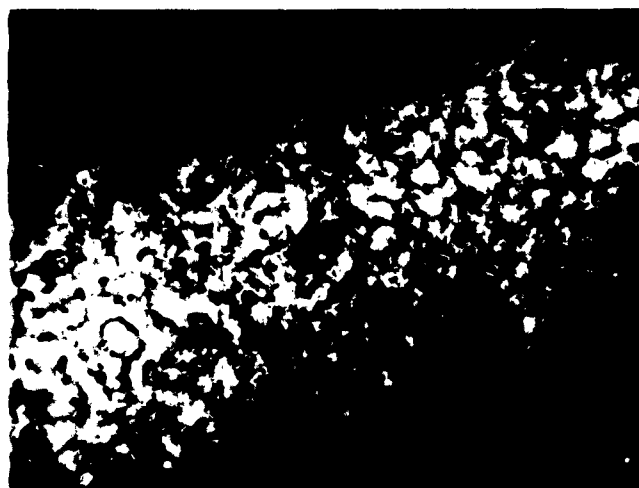


Figure 9

Transmission electron micrograph of 1500A Ge film J392 peeled from CaF_2 , 35,000X.

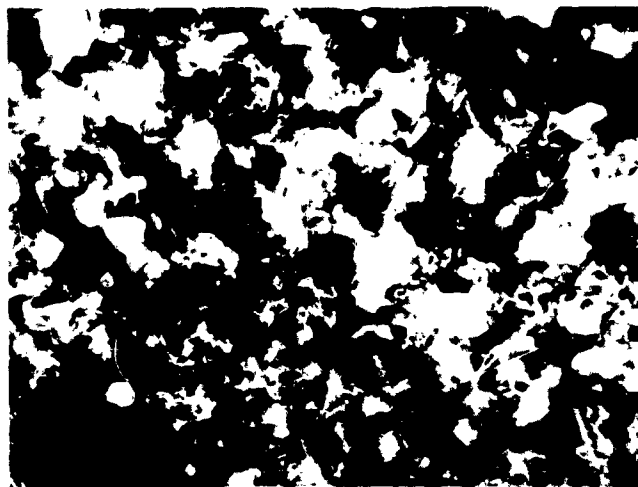


Figure 10 (a)

Transmission electron micrograph of 1500Å GaAs film J410
peeled from CaF₂, 35,000X.

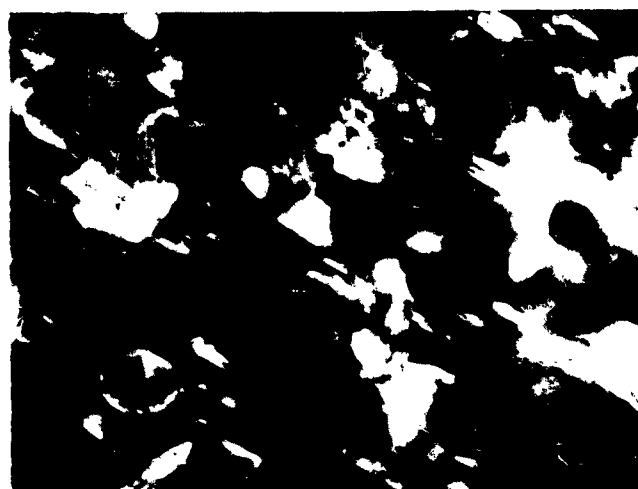


Figure 10 (b)

90,000X.

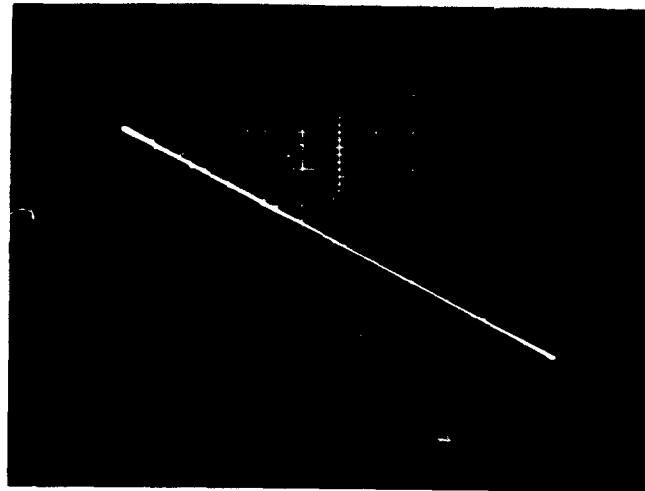


Figure 11

I-V characteristic of Si film on bulk Si with evaporated, then alloyed, contacts of Al and Au-Sb. 0.05 ma/div. vertical, 0.5 v/div. horizontal. J534.

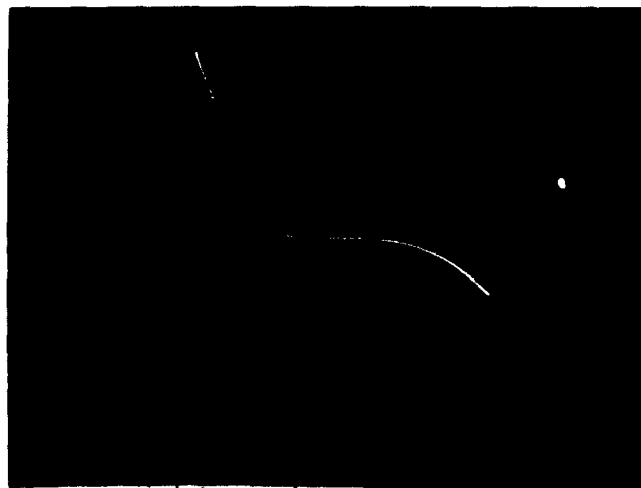


Figure 12

I-V characteristic of 5000Å p type Si film on BeO, oxidized one hour at 900°C. 6 square mil Au vs. 2000 square mil Al contacts. 0.05 ma/div. vertical, 2 v/div. horizontal. J539.

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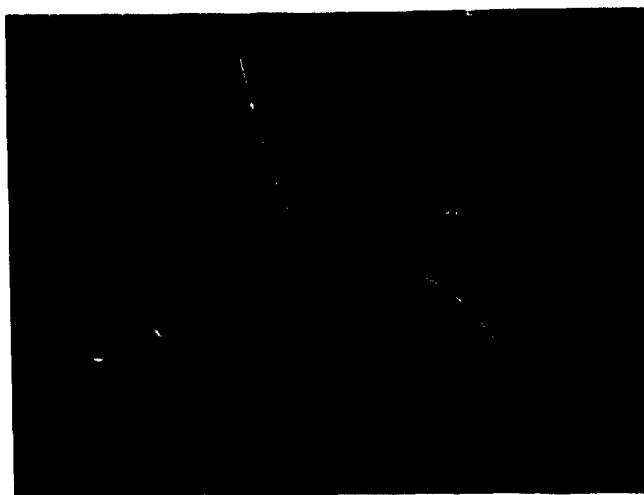


Figure 13

I-V characteristic of 5000Å n type GaAs film on CaF_2 , oxidized one hour at 550°C. 6 square mil Sn vs. 10,000 square mil Sn contacts. 0.05 ma/div. vertical, 0.2 v/div. horizontal. J530B.